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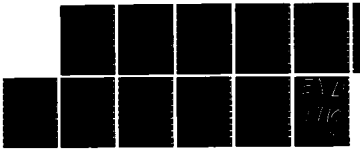

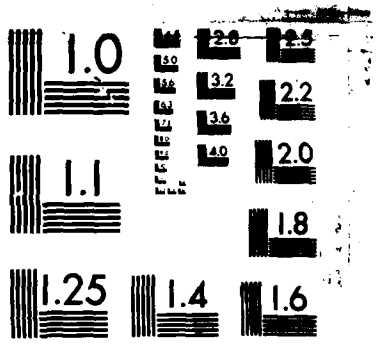

MTL TR 86-8

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THERMAL ANALYSIS OF VARIOUS EPOXY MIXTURES

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POLYMER RESEARCH DIVISION

March 1986

US ARMY
LABORATORY COMMAND
MATERIALS TECHNOLOGY
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U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
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ABSTRACT

There are three distinct types of epoxides in the SP-250 system. The epoxides were individually mixed with Monuron. Also, three-component mixtures, epoxide + Dicy/Monuron, were prepared with dicyandiamide. The mixtures were cured by differential scanning calorimetry. It was found that the isothermal heat of reactions of the two- and three-component systems were in reasonable agreement with the respective dynamic reactions, consequently, the extent of isothermal cures could be determined from the two heating modes.

The synergistic effect of dicyandiamide and Monuron, on the curing process, was simulated by the isothermal reaction of two mixtures. A type of reaction kinetics having the dimensions of temperature per time was evaluated in this report.

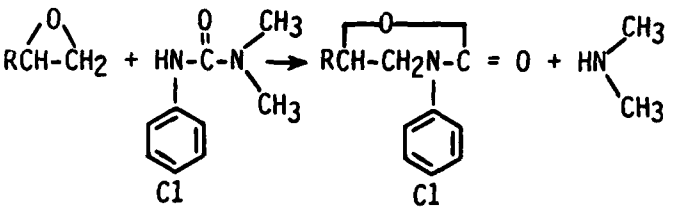
INTRODUCTION

Epoxy resins are an important class of materials that have military applications such as usage in helicopter blades. There is a continuous effort in this installation to study the curing behavior of the epoxy resins by various techniques, one of which is differential scanning calorimetry (DSC).

Previously, the various combinations which compose the SP-250 system were studied[1] by DSC, under dynamic conditions. Later, these epoxy combinations were isothermally cured for an infrared (IR) spectroscopic examination.[2] Prompted by these results, certain blends were selected for further investigation. Regarding the selected mixtures, one of the main concerns of this report is to present some of the isothermal/dynamic results originating in 1981.

RESULTS AND DISCUSSION

Dicyandiamide (Dicy) is a high temperature hardening agent. The Dicy cure of epoxy resins is dramatically accelerated by a class of trisubstituted urea derivatives such as Monuron. The accelerator (urea) undergoes a cyclocondensation reaction with the epoxy ring to form a 2-oxzolidone derivative of this type.



The cure mechanism was elucidated in this laboratory[3] some time ago. Although dimethylamine is able to slowly harden resins at ambient temperatures, the critical role of the amine on the curing process is its enhancement of the reactivity of Dicy. A small amount of dimethylamine accelerates the Dicy cure. In a more recent study, an epoxy resin and Dicy were formulated with various trisubstituted ureas so that the isothermal cures afforded two glass transitions (T_g). It was found that the lower transition temperature resulted from the high concentration of the urea derivatives.

The resin + Dicy mixture yielded a higher T_g than the resin + Dicy/urea system. Over a broad stoichiometric range for a given resin + Dicy/Monuron system, the kcal per epoxy mole is fairly constant, and this is advantageous in regard to DSC analysis. We merely wish to say that the heat of reaction of the resin + Dicy system was elusive to DSC analysis, although the mixing procedure resulted in small particle size.

1. LaLIBERTE, B. R., *The Dicyandiamide Cure of SP-250 Epoxy Resin Accelerated by Monuron.* U.S. Army Materials Technology Laboratory, AMMRC TR 83-17, April 1983.
2. SACHER, R. E., and LaLIBERTE, B. R. *Infrared Spectroscopy Epoxy Resin System.* U.S. Army Materials Technology Laboratory, AMMRC TR 84-29, July 1984.
3. LaLIBERTE, B. R., BORNSTEIN, J. and SACHER, R. E. *Cure Behavior of an Epoxy Resin - Dicyandiamide Accelerated by Monuron.* American Chemical Society, I&EC Product Research & Development, June 1983, p. 261-262.

The relationship between the exothermic reaction temperature and the heating rate for a series of DSC runs is expressed[4] below.

$$\text{Log } \phi = A/T + B$$

where:

ϕ = heating rate (^{o}C/min) assigned to a sample,
A = constant, related to activation energy (E_a),
B = constant, related to Arrhenius frequency factor, and
T = reaction temperature (^{o}K) of the sample is taken at the apex of the exotherm where maximum heat flow occurs.

Contrary to the literature,[5] the heat of reaction of diglycidyl ether of bisphenol-A (DGEBA) and Monuron is not small but in reality quite substantial. The mixture containing 2 moles of DGEBA and 0.214 mole of Monuron analyzed on DuPont 1090B Thermal Analyzer produced a value of 21.0 kcal per epoxy mole. The heating curve recorded with a heating rate of 0.5^{o}C/min was considerably more symmetrical than the one measured at an especially slow rate of 0.2^{o}C/min. The two analyses afforded a constant heat of reaction. Curing at a higher reaction temperature with a heating rate of 2^{o}C/min dramatically reduced the caloric value by about 70%. An encapsulated sample treated with a heating rate of 2^{o}C/min did not effect the reaction temperature, however, the heat of reaction was increased very significantly as compared to the open pan analysis (2^{o}C/min). The rapidity in which dimethylanine escapes from its environment is an important consideration in measuring the heat of reactions.

The SP-250 formulation is summarized in Table A.

Table A. THE SP-250 EPOXY SYSTEM

Reactants	Wt%
ECN 1273	45.8
EPON 828	38.1
Diluent	4.7
Dicy	7.51
Monuron	3.79
	99.90

The SP-250 resin system manufactured by the 3M Company of St. Paul, MN, is predominantly composed of two epoxides, one of which has an epoxy cresol Novalac structure, ECN 1273. The other resin is EPON 828, impure diglycidyl ether of bisphenol-A (liquid). Included is a small amount of an active diluent, described as a liquid flexibilizer having one epoxy group per molecule. Liquid chromatographic analysis conducted in this laboratory indicated that the diluent is a mixture consisting mainly of two epoxide ingredients.

4. CARPENTER, J. F. *Quality Control of Structural Nonmetallics.* Contract N00019-76-C-0138, manuscript prepared for the Naval Air Systems Command, Washington, DC, 14 October 1976.
5. SON, P., and WEBER, C. D. *Some Aspects of Monuron-Accelerated Dicyandiamide Cure of Epoxy Resins.* J. Applied Polymer Science, v.17, 1973, p. 1305-1313.

All possible epoxy combinations[1] in Table A afforded linear Arrhenius plots, (log ϕ versus $1/^{o}K$). The computer program calculated the A and B constants for each epoxy blend. Considering the cure temperature used in practice and at this given temperature, the program determined dynamic reaction rates having the dimensions of temperature per time. We labelled these rates as DSC scanning rates. The rates reflect the rapidity in which a sample approaches maximum heat flow.

The results of the dynamic rates and the IR analysis[2] of the isothermally-cured epoxy blends were consistent with each other. Certain epoxy blends which were selected for further analysis are indicated in Tables 1 through 5.

Figure 1 - The composition of the SP-250 + Monuron mixture is 95.90% of the epoxides and 4.10% of the urea by weight. The analysis conducted with a heating rate of $0.5^{o}C$/min produced a heat of reaction of 73.5 cal/g. In contrast, the faster rate ($2^{o}C$/min) decreased the caloric value to 20.6 cal/g.

Figure 2 - The SP-250 + Dicy/Monuron system (Table A), cured at $130^{o}C$, resulted in a heat of reaction (q_{iso}) of 100.9 cal/g. The isothermal reaction ceased in about 35 minutes.

Figure 3 - The dynamic reaction of the SP-250 + Dicy/Monuron system measured at $2^{o}C$/min afforded a caloric value (q_{dyn}) of 102.4 cal/g.

The caloric agreement between the two heating modes makes it possible to calculate the isothermal yields of samples that did not realize total cure. In order to calculate the extent of isothermal reaction there must be a reasonable agreement between the isothermal and dynamic environments as indicated in Figures 2 and 3.

$$\% \text{ Isothermal Cure} = q_{iso}/q_{dyn} \times 100. \quad (1)$$

Description of Tables 1 Through 5

All of the isothermal analyses were conducted with 2.5 hours of heating. In Tables 1 through 4 analytical samples were taken from 5 g batches. The first column denotes the cure temperature (^{o}C) of the sample. The second column indicates the time of maximum heat flow (min). The following column is the heat of reaction expressed in cal/g. The extent of the isothermal reaction (%) was calculated from Equation 1. The last column is the reaction temperature of the isothermally cured sample, measured at $0.5^{o}C$/min with the exception of Table 5 ($2^{o}C$/min). When a sample proceeds to completion there is no reaction temperature, therefore, there is no residual heat. N.D. means not determined. The heat of the dynamic cure and its reaction temperature are shown at the bottom of the tables.

Most of the heating curves of Tables 3 and 5 were lost. Fortunately the results were tabulated, however, without recording the time of maximum heat flow. Other methods to calculate yields will be discussed at the appropriate occasion.

The composition of the EPON 828 + Monuron mixture (Table 1) consisted of 95.90% of resin and 4.10% of the trisubstituted urea. The q_{dyn} value ($0.5^{o}C$/min) is 94.3 cal/g with a reaction temperature of $109^{o}C$. The reaction temperatures of $71^{o}C$ and $75^{o}C$ afforded exotherms that had a continuous upward slope, therefore, within 2.5 hours no maximum heat flow occurred. The cures conducted at $87^{o}C$ and $90^{o}C$ afforded residual material having a reaction temperature of $80^{o}C$. This temperature is $29^{o}C$

lower than the dynamic cure temperature (109°C). The pronounced decrease is most likely attributed to the low temperature glass transition (T_g). The DGEBA + Monuron cure has a T_g of about 75°C.

Table 2 depicts the cure behavior of the three-component system, EPON 828 + Dicy/Monuron. The respective amounts of reactants are 88.69, 7.52, and 3.79%. As compared to Table 1, the isothermal synergy between Monuron and Dicy commences at low temperatures. It had been previously predicted[1,2] that the presence of Dicy enhances the cyclocondensation reaction that liberates dimethylamine.

Table 3, also predicted, was the results between Tables 1 and 3. The EPON 828 + Monuron system appears to be more reactive than the SP-250 + Monuron mixture which was not completely cured at 93°C.

The DSC rates and IR analysis inferred that the diluent had an adverse effect on the resin, EPON 828 (Table 4). The EPON 828/diluent + Monuron mixture has a composition of 85.37% resin, 10.53% diluent, and 4.10% of the urea accelerator. The caloric value is 55.3 cal/g. Total reaction was not obtainable isothermally. The advancement of the reaction increased the residual temperature from 80 to 106°C. The higher temperature may reflect more of a strong resistance against total reaction than that of a low glass transition.

A diluent + Monuron mixture, having a respective composition of 95.92% and 4.08%, illustrated a bimodal heating cure (0.5°C/min). The larger exotherm had a reaction temperature of 125°C. The q_{dyn} value recorded was 13.7 cal/g. This small value may be partially contributed to the monofunctionality of the epoxides. A meaningful relationship between the isothermal cure and the dynamic reaction was not obtainable in open pan analysis.

The SP-250 resin (100 g) was slowly heated to 80°C. A powdered blend of Dicy and Monuron (12.8 g) was added under the conditions necessary to maintain the 80°C temperature (Table 5). The weights adhered to the formulary integrity of Table A. The mixing technique and preparation time are not known. The technician is presently retired.

The results of numerous analyses conducted by taking aliquots from this batch eventually lead to the determination of the isothermal cure kinetics of a structural material, the SP-250 prepreg which contains 32% reactants (Table A) and 68% glass fibers.

It is well known that the isothermal baseline is more arbitrary than the establishment of a dynamic baseline. After finding that $q_{iso} \cong q_{dyn}$, the difficulty with the baseline may be circumvented, as it is not necessary to record the isothermal heat. For example, in Table 5 the 83°C cure afforded (2°C/min) a residual heat of 91.8 cal/g ($q_{iso,dyn}$).

$$\% \text{ Isothermal Cure} = (q_{dy} - q_{iso,dyn})/q_{dy} \times 100. \quad (2)$$

The dynamic analysis of the SP-250 prepreg did not yield constant heat of reactions because of the variation in composition between the glass and the organic materials. The isothermal heat of reaction (q_{iso}) plus the residual heat ($q_{iso,dyn}$) was taken as the total heat (q_t) of the cure.

$$\% \text{ Isothermal Cure} = q_{iso}/q_t \times 100 \quad (3)$$

where:

$q_t = q_{iso} + q_{iso,dyn}$.

The data collected[6] (Equation 3) for the SP-250 prepreg received a stringent analysis according to a kinetics method of Ryan and Dutta. The fit to these equations substantiated the significance of the isothermal and dynamic measurements. Also, the prepreg-isothermal cure kinetics[6] were found to correlate with the compositional parameters obtained from high performance liquid chromatography.

EXPERIMENTAL

The preparation of the mixtures has been described.[1] The DSC analysis was conducted in open aluminum pans with the DuPont 990 Thermal Analyzer fitted to a 902 DSC plug-in module under a static atmosphere. Calibration of the instrument and establishment of an isothermal baseline has been described.[6] The areas of the exotherms were digitized using a Hewlett Packard 9830 Computer. Sample weight (10 to 20 mg) loss was negligible.

ACKNOWLEDGMENT

Appreciation is given to Mr. A. W. Reppucci for the preparation of the large batch. This report is dedicated to Mrs. C. S. LaLiberte for various reasons.

6. HAGNAUER, G. L., LaLIBERTE, B. R., and DUNN, D. A. *Isothermal Cure Kinetics of an Epoxy Resin Pregreg.* American Chemical Society Symposium, Series 221, Division of Organic Coatings and Plastic Chemistry. Las Vegas, NV, March 28-April 2, 1982. Library of Congress, (ACS Series; ISSN 0097-6156; 221).

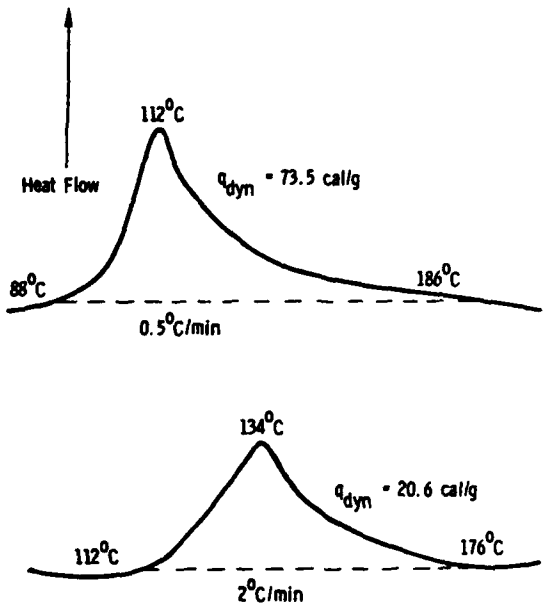


Figure 1. SP-250 + Monuron.

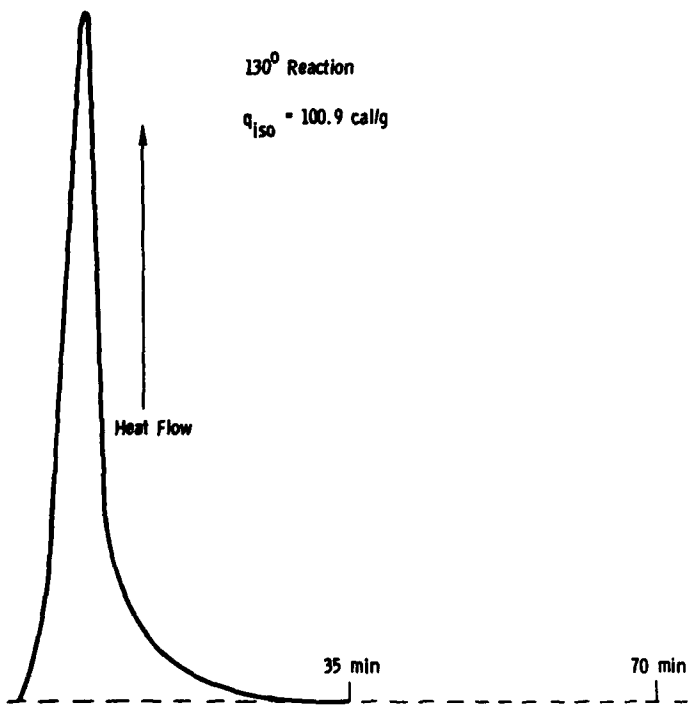


Figure 2. SP-250 + Dicy/Monuron

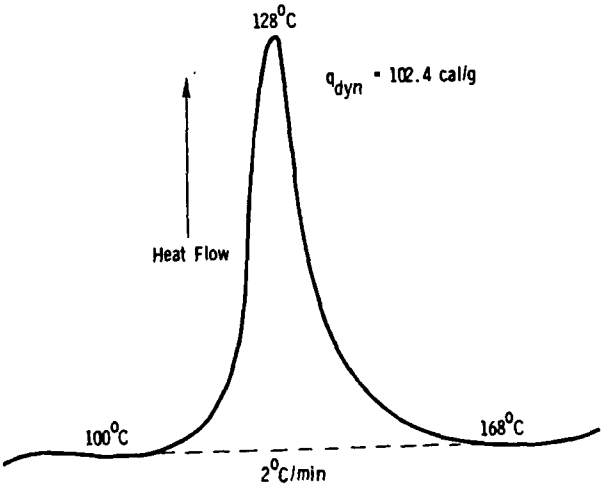


Figure 3. SP-250 + Dicy/Monuron

Table 1. THERMAL ANALYSIS - THE EPON 828 + MONURON REACTION

Iso Temperature (°C)	Apex (min)	Iso Heat (cal/g)	Iso Yield (%)	Residual Temperature (°C)
71		3.1	3.3	N.D.
75		16.6	17.8	N.D.
80	130.0	29.6	31.7	N.D.
87	67.0	63.5	68.0	80
90	63.0	68.2	73.0	80
93	13.5	93.2	99.8	No Heat
130	8.0	91.9	98.4	No Heat

Note: q_{dyn} = 93.4 cal/g; R.T. = 109°C (0.5°C/min)

Table 2. THERMAL ANALYSIS - THE EPON 828 + DICY/MONURON REACTION

Iso Temperature (°C)	Apex (min)	Iso Heat (cal/g)	Iso Yield (%)	Residual Temperature (°C)
71	113.0	61.4	N.D.	N.D.
72	88.0	73.9	N.D.	N.D.
77	48.0	97.4	N.D.	N.D.

Note: The q_{dyn} value not determined

Table 3. THERMAL ANALYSIS - THE SP-250 + MONURON REACTION

Iso Temperature (°C)	Apex (min)	Iso Heat (cal/g)	Iso Yield (%)	Residual Temperature (°C)
80		3.5	4.8	
83		7.1	9.7	
88		34.1	46.4	
90		39.9	54.3	
93		51.2	69.7	87
93		51.9	70.6	87
130	7.0	71.5	97.3	No Heat

Note: q_{dyn} = 73.5 cal/g; R.T. = 112°C (0.5°C/min)

Table 4. THERMAL ANALYSIS - THE EPON 828/DILUENT + MONURON REACTION

Iso Temperature (°C)	Apex (min)	Iso Heat (cal/g)	Iso Yield (%)	Residual Temperature (°C)
93	80.0	42.2		82
130	63.0	49.8		106

Note: q_{dyn} = 55.3 cal/g; R.T. = 109°C (0.5°C/min)

Table 5. THERMAL ANALYSIS - THE SP-250 + DICY/MONURON REACTION

Iso Temperature (°C)	Apex (min)	Iso Heat (cal/g)	Iso Yield (%)	Residual Temperature (°C)
80		11.2	10.9	120, 2°C/min
83		21.8	21.3	120, 2°C/min
88		50.6	49.4	120, 2°C/min
93		75.3	73.5	120, 2°C/min
130	6.0	100.9	98.5	No Heat
130	7.0	97.6	95.3	No Heat

Note: q_{dyn} = 102.4 cal/g; R.T. = 128°C (2°C/min)

The nomenclature assigned to the various columns is described in the Results and Discussion section.

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There are three distinct types of epoxides in the SP-250 system. The epoxides were individually mixed with Monuron. Also, three-component mixtures, epoxide + Dicy/Monuron, were prepared with dicyandiamide. The mixtures were cured by differential scanning calorimetry. It was found that the isothermal heat of reactions of the two- and three-component systems were in reasonable agreement with the respective dynamic reactions, consequently, the extent of isothermal cures could be determined from the two heating modes. The synergistic effect of dicyandiamide and Monuron, on the curing process, was simulated by the isothermal reaction of two mixtures. A type of reaction kinetics having the dimensions of temperature per time was evaluated in this report.

U.S. Army Materials Technology Laboratory,
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Bernard R. LaLiberte and Robert E. Sacher

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